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Short communication

Characterization and recycling of waste water from guayule latex extraction

*T.A. Coffelt**, *C.F. Williams*

USDA, ARS, U. S. Arid-Land Agricultural Research Center, Maricopa, AZ 85238, USA

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ABSTRACT

Guayule commercialization for latex production to be used in medical products and other high value applications is now a reality. Currently, wastewater following latex extraction is discharged into evaporation ponds. As commercialization reaches an increased scale, the liquid waste stream from latex extraction will become an environmental and cost issue. The objectives of this study were to (1) evaluate the properties of the liquid waste streams after latex extraction, (2) determine if the waste liquids could be reused, and (3) determine if any of the waste liquid could be recycled for latex extraction. Waste liquid was collected from three waste stream sources (W1–W3) from the pilot latex extraction facility operated during 2006 and analyzed for nutrient content, sodium absorption ratio (SAR), and pH. The SAR for W1 indicated it was acceptable for irrigation use. The high SAR values for W2 and W3 indicated they could not be used for irrigation because the high Na content would adversely affect soil properties. The N content of the waste liquid averaged about 0.2%, and the K values were between 500 and 600 ppm. The solids in W1 and the unpleasant odor characteristic of the waste liquid were overcome by lowering the pH of the waste liquid from over 10 to less than 7. Based on the SAR analyses, six treatments were developed for evaluating the potential for recycling W2 and W3 in the latex extraction process. Results showed that using the treatments comprised of the W2 and W3 waste streams recycled to the extraction process resulted in a lowered SAR of the waste liquid being discharged (26.95–10.5). Based on our results it might be possible to recover latex from the waste liquid by recycling. More importantly, none of the treatments associated with recycling reduced latex recovery. Reuse and recycling the waste liquids would reduce the amount of waste liquid discharged by 50% and the initial amount of extraction solution by 80%. In conclusion, results from this study show that (1) W1 can be used for irrigation after treatment and (2) recycling W2 and W3 for latex extraction is possible.

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1. Introduction

The United States imports more than one million metric tons of natural rubber annually at a cost of about one billion US dollars as well as over $24 billion of finished natural rubber products (Mooibroek and Cornish, 2000). All natural rubber and natural rubber products that the United States imports are from *Hevea* [*Hevea brasiliensis* (A. Juss.) Muell.-Arg.]. Allergies

* *Corresponding author*. Tel.: +1 520 316 6359; fax: +1 520 316 6330.
E-mail address: terry.coffelt@ars.usda.gov (T.A. Coffelt).
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to *Hevea* latex have become a serious health problem in the United States and Europe, especially with health care workers and patients who undergo multiple surgeries (Ownby et al., 1994). Guayule (*Parthenium argentatum* Gray) latex has been shown to be a source of circumallergenic latex for the manufacture of medical and other latex products due to its very low protein content and lack of epitopes that cross-react with latex allergy (Siler and Cornish, 1994, 1995; Siler et al., 1996).

Guayule and *Hevea* are the only plants currently grown commercially for natural rubber. The genus *Parthenium* is a member of the family Asteraceae and is native to most of North America (Whitworth and Whitehead, 1991; Foster and Coffelt, 2005; Ray et al., 2005). Guayule has been evaluated in the U.S. as a potential commercial rubber crop during at least three previous periods (Whitworth and Whitehead, 1991; Foster and Coffelt, 2005; Ray et al., 2005). The current commercialization effort differs from previous efforts in that the water-based extraction process produces material that does not compete with the commodity rubber markets, since the primary product is latex rather than rubber.

At the time of this study, commercialization had progressed to the construction of a pilot plant for latex extraction and the planting of over 2000 ha. Currently, a larger four story pilot plant is in operation. Wastewater following latex extraction is discharged into evaporation ponds. As commercialization reaches full scale, the liquid waste stream from latex extraction will become an increasing disposal problem. Four potential problems were identified with the disposal method used in the study pilot plant: (1) an unpleasant odor due to the high ammonia concentration of the waste liquid, (2) negative effects on soil properties due to the high Na concentration in the ammoniated antioxidant solution (AAO) used in the extraction process, (3) potential loss of latex in the waste liquid, and (4) potential treatment and disposal costs of the waste water to meet regulatory guidelines. Ammonia is also used in the current pilot plant but not in the extraction medium itself. In addition, either the potential negative effects on soil properties and/or the presence of latex in the wastewater could slow the evaporation process and infiltration of water into the soil. The very slow infiltration of water into the soil profile has been observed in the current evaporation ponds. The objectives of this study were to (1) evaluate the properties of the liquid waste streams after latex extraction, (2) determine if the waste liquid could be reused, and (3) determine if any of the waste liquid could be recycled for latex extraction.

2. Materials and methods

2.1. Waste stream evaluation

Waste liquid was collected from three waste stream sources in the pilot latex extraction facility (Fig. 1). In Objective 1, each of the three sources was analyzed for Na, K, Ca, Mg, and pH. The concentrations of Na, K, Ca, and Mg were determined using a flame atomic absorption spectrophotometer and standard sample preparation methods. The pH was determined using a pH meter. Sodium adsorption ratio (SAR) was calculated for each based on the Na, Ca, and Mg contents according to the following equation:

$$\mathrm{SAR} = \frac{[\mathrm{Na}^{+}]}{\sqrt{[\mathrm{Ca}^{2+}] + [\mathrm{Mg}^{2+}]}} \tag{1}$$

2.2. Potential for water reuse and/or recycling

Based on the analyses of the three wastewater sources in Objective 1, two approaches were tested for possible reuse of the waste streams. For Objective 2, the waste streams were treated with phosphoric acid to lower the pH of a subsample from each wastewater source to a pH of 6.5. Following the pH adjustment the subsample was processed through a clarifier (Westfalia model LWA205, Oelde, Germany) to remove solids from the subsample. The odor from each sample was rated by six personnel before and after the pH was adjusted to evaluate the effects of pH change on odor. The odor of the wastewater prior to treatment is very distinctive. Each participant rated the odor as noticeable or not noticeable before and after pH treatment. The amount of solids removed by the clarifying step was visually evaluated to determine the suitability of the remaining liquid for water reuse and/or recycling in the latex extraction process.

As a result of this evaluation, six treatments were developed for evaluating the potential for recycling two of the waste streams (W2 and W3) in Objective 3. Treatment 1 was a control using 100% original/new ammoniated antioxidant solution for each extraction. Treatment 2 was a mixture of 70% original and 30% from the final washing step (W3) (Fig. 1). Treatment 3 was a mixture of 50% original, 30% W3, and 20% from the initial washing step (W2) (Fig. 1). Treatment 4 was a mixture of 20% original, 30% W3, and 50% W2. Treatment 5 was 100% W2. Treatment 6 was 100% W3. All mixtures were based on the volume percent of the final mixture. The pH of the two wastewater sources used was not adjusted prior to use in the treatment mixtures and they were not clarified prior to use in the treatment mixtures. After the mixtures for the six treatments were made, each was analyzed for Na, K, Ca, Mg, C, and N. The nutrient contents for Na, K, Ca, and Mg and the SAR values were determined as in objective 1. The C and N contents were determined by a Shimadzu total organic carbon and nitrogen analyzer (Shimadzu Scientific Instruments Inc., Columbia, MD, USA).

For evaluating the six treatments for potential recycling during the latex extraction process, 1 kg of partially defoliated shrub material obtained from the pilot plant was chipped using the method described by Coffelt and Nakayama (2007). The six treatments were used to replace the initial antioxidant solution in the chipping method described by Coffelt and Nakayama (2007). The six antioxidant treatments were added to the chipped material immediately following chipping to give approximately a 1:1 ratio of plant material to antioxidant solution by weight. The laboratory latex extraction process of guayule shrub described by Cornish et al. (1999) was used to complete the latex extraction process. The six treatment solutions were used during the laboratory extraction phases to replace the ammoniated antioxidant solution normally used by the study pilot plant. The amounts of solution used were not altered. Latex concentrations and total latex were determined on a dry weight basis. Following latex extraction, the

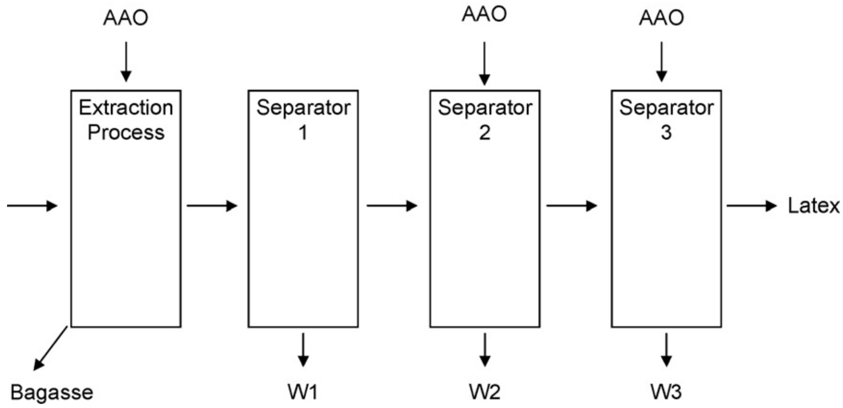

Fig. 1 – Diagram of the latex extraction process with collection sites within the process of the three sources of wastewater (W1, W2, and W3), and sites where the ammoniated anti-oxidant solution (AAO) was added during the latex extraction process.

homogenate from each sample was analyzed for Na, K, Ca, Mg, SAR, C, and N as described above. In addition, the electrical conductivity (EC) value was determined. Each treatment was replicated four times in a completely randomized design. Data were analyzed by analysis of variance and means separated by LSD at the 0.05 level.

3. Results

3.1. *Analyses of three waste streams*

Results from analyses of the three waste stream sources (Table 1) showed that all waste sources were high in Na (>300 ppm) and the pH was about 11 as expected. We did not expect the high values for K, Ca, and Mg in W1, especially the very high K values (>900 ppm). These values decreased from W1 to W3 indicating that they were probably being extracted from the plant material and removed from the latex enriched streams during the extraction and washing process. The high SAR values for W2 and W3 indicate that these two waste streams would not be recommended for reuse as irrigation water due to the dominance of sodium in solution.

3.2. *Potential for water reuse*

Initial treatment of the waste streams for potential water reuse requires neutralization of the high pH. The pH was lowered by titrating with phosphoric acid to a pH of 6.5. A subsample of each solution was passed through a clarifier following pH neutralization to remove any solids. Adjusting the pH and processing the three waste sources through a clarifier accomplished two goals. The first was removal of the objectionable odor from the waste liquid. All six personal rated the odor noticeable before pH adjustment and not noticeable after the pH adjustment. Second was precipitation of the solids (plant material and soil) from the waste liquid. The amount of precipitate was visually much greater in W1 than in the other two sources as expected. Initial observations indicated the amount of precipitate in W2 and W3 would probably not preclude them from being used for recycling in the latex extraction process. In contrast, the large amount of precipitates in W1 appeared to be sufficient to exclude W1 for consideration for recycling into the initial extraction process. However, two simple steps, lowering the pH followed by removal of the solids, would make the W1 waste stream acceptable for water reuse.

3.3. *Use of waste liquid in latex extraction treatments*

The results from the initial analyses of the six treatment solutions developed from W2 and W3 were similar to the three original sources for Na (Tables 1 and 2). The K, Ca, Mg, and SAR values varied according to the percentage of each wastewater source in the treatment. Initial SAR values for all treatments (Table 2) would make them unacceptable for use as irrigation water as was shown in the initial analyses of W2 and W3 (Table 1). The results from these analyses also indicate that the high Na value in the waste liquid streams is from the original antioxidant solution (Treatment 1) used in the extraction process, while the high K, Ca, and Mg values are from the extracted plant material.

Table 1 – Results obtained for sodium (Na), potassium (P), calcium (Ca), magnesium (Mg), SAR values, and pH of waste liquid from three sources in a pilot guayule latex extraction facility.

Source	Na (ppm)	K (ppm)	Ca (ppm)	Mg (ppm)	SAR	pH
W1	325	946	185	61	8	10.7
W2	422	225	46	17	19	11.2
W3	360	49	9	3	38	11.6

Table 2 – Results from the initial analyses prior to latex extraction of the six recycling treatments used in a guayule latex extraction experiment for sodium (Na), potassium (K), calcium (Ca), magnesium (Mg), and SAR value.

Treatment[a]	Na (ppm)	K (ppm)	Ca (ppm)	Mg (ppm)	SAR
T1	404	0.4	0.6	0.1	176
T2	393	9.1	2.2	1.1	76
T3	421	22.9	9.7	5.9	37
T4	413	81.7	20.4	7.2	28
T5	390	155.0	37.0	16.0	19
T6	454	28.4	7.9	3.1	49

[a] T1 = 100% original ammoniated antioxidant solution (OAAO); T2 = 70% OAAO + 30% W3; T3 = 50% OAAO + 30% W3 + 20% W2; T4 = 20% OAAO + 30% W3 + 50% W2; T5 = 100% W2; T6 = 100% W3.

Comparison of the waste solution from the six treatments after latex extraction showed Na and N contents of the five recycle treatments (2–6) were not significantly different from Treatment 1, the original antioxidant extraction solution (Table 3). The K content was significantly lower for Treatment 1 than Treatments 4 and 5. Treatment 1 was significantly lower in Ca than Treatments 3–6. Treatment 6 was significantly higher in Mg content than Treatments 1 and 2. Carbon was significantly higher in Treatments 5 and 6 than Treatment 1. SAR was significantly lower for Treatments 3–6 than Treatments 1 and 2, while EC was the reverse. These results agree with the initial evaluations (Table 2) which showed during the latex extraction that large amounts of K, Ca, and Mg are being extracted from the guayule plant material.

The amount of latex extracted using the recycle treatments was significantly higher for Treatments 4 and 5 than Treatments 1 and 2 (Table 4). This is likely a result of additional latex recovery from the wastewater and not extraction efficiency or additional latex being extracted from the plant material using these treatments. The increased latex seemed to be related more to the percentage of the treatment solution that was W2 than other factors, since Treatment 5 was 100% W2 and Treatment 4 was 50% W2.

The actual amounts of latex extracted in this experiment for Treatment 1 (2.89%) were lower than expected based on previous results from the pilot plant (Cornish, personal communication, 2008), but similar to those in previous studies (Coffelt et al., 2005, in press-a,b; Dierig et al., 2001). The low amount of latex extracted in this and other studies may be due to dehydration of the guayule material selected for processing (Coffelt et al., in press-a). In spite of the low latex concentration in the original guayule shrub material, the results show that using W2 and/or W3 for recycling did not adversely affect latex extraction.

Table 4 – Total guayule latex extracted from 1 kg samples using nine recycled antioxidant solutions compared to a non-recycled control treatment.

Treatment[a]	Total latex (%T1)
T1	100 c[b]
T2	100 c
T3	142 bc
T4	240 ab
T5	279 a
T6	157 abc

[a] T1 = 100% original ammoniated antioxidant solution (OAAO); T2 = 70% OAAO + 30% W3; T3 = 50% OAAO + 30% W3 + 20% W2; T4 = 20% OAAO + 30% W3 + 50% W2; T5 = 100% W2; T6 = 100% W3.
[b] Means followed by the same letter are not significantly different according to Fisher's LSD at $P = 0.05$.

4. Discussion

Reducing the pH and clarification of the waste streams corrected two of the problems associated with the waste streams—the unpleasant odor and suspended solids of the discharged water. A pH of 6.5 also resulted in coagulation of residual latex capable of coating the soil surface with a hydrophobic layer that could reduce water infiltration into the soil. If the pH is not lowered to <7, then discharge of water with a pH >11 will disperse all of the soil organic matter and desta-

Table 3 – Nutrient contents of the homogenate for sodium (Na), potassium (P), calcium (Ca), magnesium (Mg), N (nitrogen), and carbon (C), and SAR and EC values for six antioxidant solutions used in guayule latex extraction.

Treatment[a]	Na (ppm)	K (ppm)	Ca (ppm)	Mg (ppm)	N (ppm)	C (ppm)	SAR	EC (dS/m)
T1	377.0 ab[b]	507.0 c	79.0 c	24.0 bc	1852.0 ab	3615.0 cd	14.0 a	0.570 b
T2	379.0 ab	537.0 bc	86.0 bc	20.0 c	1592.0 b	3337.0 d	14.0 a	0.608 b
T3	355.0 b	591.0 abc	116.0 ab	32.0 ab	1810.0 ab	3907.0 bc	11.0 b	0.663 a
T4	360.0 b	610.0 ab	122.0 a	35.0 ab	1760.0 ab	4160.0 abc	11.0 b	0.693 a
T5	366.0 ab	645.0 a	135.0 a	34.0 ab	1893.0 a	4562.0 a	10.0 b	0.708 a
T6	390.0 a	567.0 abc	124.0 a	36.0 a	1758.0 ab	4165.0 ab	11.0 b	0.693 a
LSD	29.0	89.0	27.0	11.0	287.0	483.0	1.6	0.052

[a] T1 = 100% original ammoniated antioxidant solution (OAAO); T2 = 70% OAAO + 30% W3; T3 = 50% OAAO + 30% W3 + 20% W2; T4 = 20% OAAO + 30% W3 + 50% W2; T5 = 100% W2; T6 = 100% W3.
[b] Means followed by the same letter within columns are not significantly different according to Fisher's LSD at $P = 0.05$.

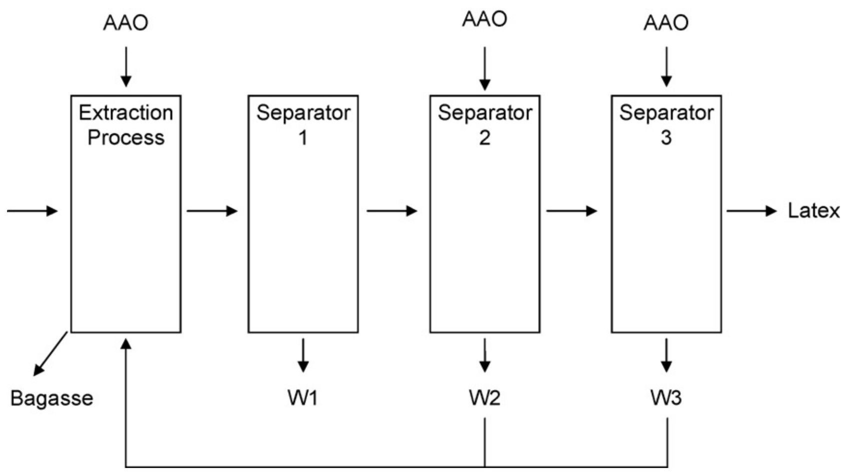

Fig. 2 – Diagram of a latex extraction process that recycles 100% of W2 and W3. The flow of ammoniated anti-oxidant solution (AAO) is reduced to accommodate the flow of W2 and W3 into the process.

bilize the natural soil structure. Therefore, pH neutralization is necessary for discharge and potential reuse of the water for irrigation or other uses. A treatment scheme of pH neutralization followed by sedimentation and surface removal of floating latex will remove the odor and solids associated with the waste liquid.

The remaining nuisance of Na-induced dispersion of the soil surface can be mitigated through the recycling of W2 and W3 and only discharging W1 (Fig. 2). Results showed that using any of the treatments to recycle waste liquid resulted in a lower SAR of the waste being discharged (26.95–10.5). Ayers and Westcot (1989) have shown that SAR and EC are indications of when water should not be used for irrigation (Table 5). In combination, these values are an indication of when soil structure can be lost due to imbalances in the ions in the soil and can also be an indication of when salt sensitive or semi-tolerant crops cannot be grown (Table 5). For example, the high SAR values (19–38) we found in W2 and W3 (Table 1) would make this waste liquid unacceptable for use as irrigation water regardless of the EC due to the potential damage on plants at these SAR values. Use of W2 and W3 on soils would have negative effects on soil properties, while the SAR value of 8 for W1 would be acceptable as long as the EC value was greater than 0.5. The SAR values of 10–11 and EC values of 0.6–0.7 show that the waste streams from Treatments 3–6 following recycling (Table 3) would be acceptable for use as irrigation, while Treatments 1 and 2 with SAR values of 14 and EC values of 0.6 would not be acceptable. Based on these results, we propose that W1 with pH adjustment below 7 and removal of solids could be used for irrigation.

The N content of the waste liquid averaged about 0.2%. Thus, another possible use of the waste liquids would be as fertilizer. The potential use as fertilizer is also indicated by the high K values (500–600 ppm). The high K values also indicate that guayule probably has a high K requirement and may require extra K fertilizer especially where soil tests are low in K. If the solids in the waste stream and the obnoxious odor characteristic of the waste liquid are overcome by lowering the pH of the waste liquid from over 10 to less than 7 with phosphoric acid, then the waste solids would be high in N, P, and K and may be an acceptable fertilizer for plant use.

The results for W2 and W3 in the recycling study show that the waste liquid obtained during latex extraction can be recycled for latex extraction, thus, reducing the amount of waste liquid discharged by 50%. Recycling the waste liquid would also reduce extraction costs by substituting the recycled waste liquid for 80% of the extraction solution. Recycling waste liquid would also reduce the total amount of waste liquid to be disposed, thus, reducing treatment and disposal costs. None of the treatments associated with recycling

Table 5 – SAR and EC values of irrigation water and their interaction that may result in plant growth and/or soil infiltration problems based on results from Ayers and Westcot (1989).

SAR		Effects on soil properties		
		None	Slight to moderate	Severe
0–3	$+EC_w$	>0.7	0.7–0.2	<0.2[a]
3–6	$+EC_w$	>1.2	1.2–0.3	**<0.3**
6–12	$+EC_w$	>1.9	1.9–0.5	**<0.5**
12–20	$+EC_w$	**>2.9**	2.9–1.3	**<1.3**
20–40	$+EC_w$	**>5.0**	**5.0–2.9**	**<2.9**

[a] Values in bold indicate SAR and EC value combinations that are detrimental to plant growth.

reduced latex recovery. Increases in latex are probably due to recovery of latex from the waste liquid rather than increased extraction from the shrub as indicated by similar changes in SAR values between the treatments. If the increase was from increased extraction, then one would expect the SAR values to change.

Based on our results, we recommend the waste liquids W2 and W3 be recycled and directly added to the ammoniated antioxidant solution, solution used in the initial extraction process. The current processing facility has the capability to recycle these waste streams. The waste liquid W1 should be pH adjusted to 6.5 and clarified to remove solids and odor prior to disposal. The liquid portion could be used as a fertilizer, disposed of by evaporation ponds or irrigation, or used to keep unprocessed shrub moist until processed (Coffelt et al., in press-a). The solid portion could be used as mulch or an organic amendment. These uses would need additional research to determine the best use. Areas that should be investigated further are (1) the quality of the latex being recovered and (2) the changes recycling W2 and W3 would have on the waste streams after recycling is started.

5. Conclusions

Results from this initial study indicate that the four potential problems associated with the waste streams generated during guayule latex production can be overcome relatively easy and may result in potential beneficial use of the waste streams. The unpleasant odor of the waste streams can be corrected by lowering the pH to <7. The potential negative effects of high Na and ammonia concentration can be overcome by recycling W2 and W3 in the extraction process, and pH adjustment followed by clarification of W1. Following pH adjustment and clarification, W1 can be used for irrigation. Recycling W2 and W3 may allow recapture of any latex in these waste streams as they are returned to the extraction process. Recycling of both W2 and W3 would also reduce the amount of liquid waste by about 50% and could replace about 80% of the initial extraction solution used during the first processing step.

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